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CONTRACTOR REPORT ARCSL-CR-77046

OPTIMIZATION OF THE MATERIAL FOR CONSTRUCTION OF THE NEW PROTECTIVE MASK

FINAL REPORT

BY

J. P. Daugherty

September 1977

GENTEX CORPORATION
Carbondale, Pennsylvania 18407

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US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
Chemical Systems Laboratory
Aberdeen Proving Ground, Maryland 21010

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1	Optical properties Physical properties Curing temperature Curing pressure Silicone elastomer	Elastomer Polyurethane Processability Contaminates	Toroidal lens Lens Outsert Brabender Rheology	Scorch rate Strata		

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PREFACE

This work was performed under Contract DAA15-75-C-0175 with Edgewood Arsenal, Aberdeen Proving Ground, Maryland, from July 1, 1975 to July 13, 1976 by Gentex Corporation, Carbondale, Pennsylvania 18407.

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SUMMARY

As part of the development of a new Face Mask, Edgewood Arsenal selected a silicone rubber as their prime candidate. This material has good clarity, flexibility and a wide temperature capability. Drawbacks are; poor scratch resistance, aging discoloration and poor impermeability. The objective of this project was to evaluate the polymer rheology, curing behavior, physical and optical properties of Dow Corning silicone X4-2665. The Toroidal lens mold was used extensively in this evaluation. Several other elastomers, such as, polyurethane, EPDM (ethylene propylene diene terpolymers) and EPR (ethylene propylene copolymers) were evaluated in an effort to improve on the barrier and abrasion resistance property deficiencies of silicone.

Initial work involved EPDM, EPR and polyurethane elastomers, but all of these materials were discarded as Face Mask candidates due to insurmountable problems with either optical quality or molded physical properties. Polyurethane (Pellethane 2363-80A) lens Outserts were molded using Gentex' visor mold as a substitution for the polyurethane Toroidal lens requirements. The silicone Toroidal lens requirements were increased to include the EPDM and flat lens requirements.

During the remainder of this project, we were able to establish processing parameters for the production of optically good Toroidal lenses with the GFM transfer mold in a Carver 100-ton press and processing the silicone on a 6-inch x 12-inch laboratory 2-roll mill.

Cure times were reduced to a minimum of four minutes with a change time of 3.5 minutes for a total cycle time of 7.5 minutes, using lot numbers 004 and 005 of Dow Corning's X4-2665 silicone. It would be possible to reduce the cure time more with heat-cored molds to reduce mold cooldown.

High reject rates were generally caused by material contamination or by improper molding conditions. Contamination rejects can be due to, or occur in, the virgin elastomer, machine or airborne and/or dirty mold. The elastomer lots from 003 - 007 that we have evaluated appeared to be very clean and were a very minor source of contamination.

In order to combat high reject rates from the remaining contaminates, there is a need for an exceptionally clean area with positive pressure and preferably laminar air flow. All precautions of extreme cleanliness should be taken including thorough vacuuming of the room and equipment followed by a damp cloth wipe. Proper clothing should be supplied including hats and gloves. A method for checking the cleanliness is to use a "black light" (ultraviolet) in a darkened area.

Flow lines and, in excessive cases, "orange peel" can be a major reject caused by improper molding temperatures and/or press pressures. Molding temperatures are critical and may have to be varied for different lots of material, age of compounded stock and/or total processing heat history of the accelerated compound.

Physical properties of different lots of X4-2665 appeared to be very consistent when post-cured. Non-post-cured slabs and lenses varied to a greater extent, mainly due to the human element in molding.

Optical properties of the material and Toroidal lenses were within acceptable limits, as viewed perpendicular to the surface. However, prismatic image displacement encountered due to as-worn position was a problem that needed a lens design modification.

Physical property studies of irradiation-treated lenses and slabs indicated that a relatively low dosage of no greater than six (6) megarads be used to cross-link and adhere the lens coatings. Higher values caused excessive degradation of the base lens material.

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I. INTRODUCTION

Considerable work has been accomplished by Edgewood Arsenal and Dow Corning in developing a new protective mask using an 'optical grade silicone elastomer. This material has a wide range of temperature capabilities, optical clarity and flexibility.

A mask design fabricated with the above material gives much greater visibility than current protective masks. This program was concerned with the material analysis and establishing processing parameters and recommendations for the molding of this mask using GFM Toroidal Lens Mold.

II. MATERIAL CANDIDATES

A. Polyurethane Elastomers

The use of polyurethane elastomers for the full face mask was jointly determined to be not feasible due to limitations in high stiffness and hardness. It is possible to reduce both of the above, but only at the excessive sacrifice of critical physical properties. Therefore, all of the following work with polyurethane was limited to the fabrication and testing of lens "Outserts".

- 1) Injection molding trials were made with Upjohn Pellethane grades 2103-80A, 2103-90A and 2103-55D in an available polycarbonate Gentex visor mold.
- 2) Results of this first trial with polyurethane material is reported below for samples as molded and post-cured 13 hours at 240°F. The total light transmittance (TLT) and haze for these parts are generally improved by postcuring.

Pellethane Grade	As Molded	Post-Cured
2103-80A		
TLT, %	86.3	88.0
Haze, %	33.5	12.7
2103-90A		
TLT, %	90.0	87.5
	(90.2)1	(88.0) Retest
2103-55D		
TLT, %	90.7	91.8
Haze, %	2.4	1.4

- 3) The 2103-80A material was submitted for hardness and stiffness evaluation only, due to poor TLT and severe haze.
- 4) The 2103-80A was determined to be the best for hardness and stiffness. Poor optical performance of this stock was attributed to high moisture content. The visor mold, scheduled for refurbishing, was returned and additional trials were made with 2103-80A. This trial was more successful and samples were delivered to Edgewood Arsenal. The improved TLT and haze properties of this run are shown below.

2103-80A	As Molded	Post-cured (14 hrs @ 240°F)
TLT, %	89.7	89.8
Haze, %	8.1	5.2

- 5) Four (4) Outsert lenses were cut from 2103-80A material (not post-cured) and given to Mr. C. Shoemaker during his visit to Gentex on September 10, 1975. These lenses were a little short on each side due to the size limitations of our visor.
- 6) We have been in contact with Upjohn and they are working with an experimental urethane grade with less haze and more light stability. They will send samples to us when available which they predict to be in 3 to 4 months.
- 7) The current 2103 grades are a polyether backbone with an aromatic isocyanate (MDI). Polyester urethanes with an aliphatic isocyanate give the best clarity and light stability, but sacrifice hydrolytic stability. This type of thermoplastic urethane is not available from Upjohn and is generally more difficult to process.

Fade-O-Meter tests were performed to check the Ultraviolet stability of urethane films and coatings. Upjohn urethane coated Toroidal lens, Wilmington, Chemical urethane coated Toroidal lens, Upjohn 2363-80A film and non-treated silicone control Toroidal lens were exposed for 220 hours in a Fade-O-Meter. Appendix Table I indicates the percent total light transmittance and percent haze from 0 to 220 hours. The Upjohn materials are definitely less stable to sunlight and Ultraviolet transmission. This is most likely due to the reasons stated in Item No. 1 above. Upjohn's urethane grades have a polyether backbone and incorporate an aromatic isocyanate where as polyesters with an aliphatic isocyanate are much more light stable.

A search was made for a source for a polyester urethane with an aliphatic isocyanate. K. J. Quinn, Malden, Massachusetts was the only source that would supply us with this material. However, after repeated trials and development effort, K. J. Quinn announced that they could not supply an 80 Shore A hardness version of this material due to their processing problems.

Urethane toxicity information was requested and is as follows:

- 1) Information on Upjohn's Pellethane 2363 which has had limited testing as implants with no adverse effects.
- 2) No such information is available for Pellethane 2103 series due to the very small amount (less than 1%) of heavy metals used as a stabilizer.
- 3) Pellethane 2363 and 2103 series are the same except for the lack of stabilizer. The effect of this was claimed to be negligible in respect to processing and ultimate physical properties. Trails with this material proved that this was so.

B. Ethylene - Propylene Elastomers

Two major sources of these base elastomers were contacted in an effort to obtain a good optical-transparent grade.

- 1) DuPont was contacted three times and were not successful in developing a satisfactory transparent ethylene-propylene terpolymer (EPDM). DuPont's interest in this program appeared to be lacking or very small. Believe that they are afraid of producing large quantities of this material that would be contaminate free.
- 2) Exxon Chemical Company was contacted and two (2) samples of compound and mixed (EPDM and EPR) were supplied to us from their best efforts to meet the optical requirements.

Mold trials with both compounds indicated severe haze and extremely poor hot and cold tear resistance.

For the above reasons and problems, it was a joint agreement between Edgewood Arsenal and Gentex Corporation to terminate this portion of the contract.

III. UNVULCANIZED RHEOLOGY

A rheology study of silicone lots 002 and 003 using a Brabender Plastic-Corder to determine minimum viscosity, scorch rate and cure rate. This, as well as other similar pieces of equipment such as Monosanto Rheometer and Moony Viscometer, should only be used as tools to indicate processability parameters and not relied upon to establish absolute cure times. Any particular processor can set-up limits for particular equipment in order to provide uniform processability and ultimately, cured products.

A. Processability Comparison of Lots 002 and 003

There was a definite difference in lots 002 and 003 supplied by Dow Corning in processing, scorch, minimum viscosity, cure rate and shelf life, as indicated below.

- 1) Processability Lot 003 has much more tack and was more difficult to handle on the mill during mixing. Its tack or tendency to stick to the mill rolls, was greater than its green strength. Lot 003 was not impossible to process, but was more difficult. This condition also made it more prone to pick-up contaminates.
- 2) Scorch Rate (Measure of processing safety) Lot 003 has from 3 to 4 minutes more processing safety as determined by our Brabender analysis. The scorch time was determined by the total time for the stock to rise three torque units above the minimum viscosity torque level. Standard test conditions used with Brabender were 50 gm charge and chamber temperature of 107°C (225°F).
- 3) Minimum Viscosity Lot 003 had a lower viscosity of 2 to 3 torque units which generally helps in improved stock transfer.
- 4) <u>Cure Rate</u> Theoretically the cure rates of lots 002 and 003 were pretty much the same once they started to cure. However, the Brabender data indicated that lot 003 was 3 to 4 minutes slower which was due mainly to the improved scorch safety. This should help stock transfer without appreciably altering cure time.

- 5) <u>Uncured Stock Shelf Life</u> As measured by scorch safety (time for 3 torque unit rise) over a period of time, lot 003 had more scorch safety after four days at room temperature (72°F) than lot 002 had after one day. This was also substantiated in molding trials.
- A. The above differences were discussed with Dow Corning personnel and are believed to be due to the following:
- 1) Lot 003 was the first production size run and the molecular weight was slightly lower than lot 002.
- 2) Lot 003 had a reduction in cross linker and inhibitor to improve tear resistance.
- 3) The combination of the above properties contributed to the differences in these two lots. It was too early to tell what the total effect would be on the ultimate product, but we would guess that it might result in slightly lower tensile strength, increased compression set, lower hardness and lower modulus.
- C) We noticed a phenomena that the scorch safety improved during one and two days after mixing. Dow Corning could give no explanation for this. Many elastomers improve in uncured properties upon standing due to "wetting action". However, this is mostly due to the incorporation of fillers and this is not the case with this particular silicone, because the fillers have been incorporated prior to blending.

IV. OPTICAL PROPERTIES

The Toroidal lens optical properties were measured both in a relaxed state and using a fiberglass/resin holder made using the male mold insert as a form. The optical measurements were made based on MIL-L-38169.

Actual test areas of the Toroidal lenses are indicated in the attached figure and found in Appendix Table II.

Data are shown in Appendix Tables III through VI.

- A. Optical measurements were taken using the following instruments:
- 1. Gardner Haze Meter Total light transmittance and haze.
- 2. Telescope Prismatic deviation, refractive power and definition.
- 3. Ann Arbor Distortion.
- B. Optical measurements were taken perpendicular to lens surface and not in the "as worn" position. A special holder is necessary for the later measurements. For the same reason, horizontal and vertical deviations could not be determined.
- C. Total transmittance for all lots was over 90 percent as measured using source "C" on the haze meter. This appears to average between 2.5 3.0 percent higher than Dow Corning's values. Difference could be due to instrument used by Dow Corning, and/or conditions of their slab mold.
- D. Percent haze varies from slightly over 3 percent to almost 5 percent with the highest being lot 32059. Lot 004 had the lowest haze.
- E. Telescope and Ann Arbor properties were generally good in the relaxed position and some distortion was noted due to stretching when the fixture was used. These properties are probably affected more by molding techniques and/or tooling than polymeric variations.
- F. Additional optical information on Lot 004 appears in the attached Appendix Table VII. I have also attached information about "Cylindrical Power" as determined by Omnitech, Inc., a subsidiary of Gentex Corporation in Appendix Table VIII. The 0.06 diopter reading (in a plano position) is not excessive. However, there is no question that problems will arise when the lens is in the "as worn" position.
- G. After 340 hours in the Fade-O-Meter, there has been no change in total light transmission or increase in haze.

Total light transmittance and haze was measured before and after 600 hours in an Atlas Fade-O-Meter using a Gardner Haze Meter. The results are shown in Appendix Table I and summarized below.

- 1. No serious degradation to the surface or internal was noted after exposure for 600 hours.
- 2. The decrease in percent TLT and increase in percent haze was either due to surface dirt from the Fade-O-Meter or possibly surface haze.
- 3. The surface haze or dirt was easily removed with detergent and water.
- H. All of the available silicone lots were evaluated using a Cary-14 Spectrophotometer over the range of 200 to 2000 non-ometers. The following observations were made:
- 1. All lots have essentially the same characteristic traces over the wave lengths tested.
- 2. All have very poor Ultraviolet and near Ultraviolet attenuation. There is a sharp break in all curves at about 280nm. Listed below are the total light transmissions at the indicated wave lengths. From this the average erythemal UV transmittance is calculated per MIL-V-43511 (3.4.6) visor specification.

Spectral Transmittance at Indicated Wave Lengths

Lot Number	-32059	001	002	003	005
(Wave Lengths,					
nm)					
250	0	0	0	0	0
270	0	0	0	0	0
290	45	50	35	34	44
300	57	58.5	46	46	54
310	65	65	56	55	61
320	70	69	62	62	65
Average % Trans	39.5	40.4	33.2	32.8	37.3

The maximum average listed in MIL-V-43511 is 1 percent.

1. In an effort to reduce haze and improve clarity, an optical brightner was incorporated into the silicone in concentrations of 0.02%, 0.1% and 0.7%, and the results are shown below.

Sample No.	% Brightner	TLT, %	Haze, %	
1	0.0	90.6	3.12	
2	0.02	86.1	4.17	
3	0.1	82.3	4.61	
4	0.7	77.3	5.90	

V. PHYSICAL PROPERTIES

A comparison was made of stress-strain, tear and compression set properties of both ASTM slabs and Toroidal lenses directly from the mold and oven post-cured.

A. Stress-Strain and Tear Resistance

The comparison values for various lots of X4-2665 material are shown in Appendix Tables X and XI for material press cured 10'/270°F and with an oven post-cure of 4 hours/350°F. This information is summarized below:

- 1. Generally the physical values of ASTM slabs and Toroidal lenses agree within experimental error.
- 2. Post-cured results tend to be more uniform and the expected differences between non-post-cured part were as expected.
 - a. Increased hardness
 - b. Increased modulii
 - c. Reduced ultimate elongation
 - d. Reduced tear resistance
- 3. There appeared to be a trend of slightly lower tear resistance with lots 004 and 005, but they are high enough values to provide a satisfactory product.

B. Compression Set B

A comparison of compression set B, no post-cure vs. post-cure of various silicone lots and the effect of post-cure time and temperature using silicone lot number 005, are shown in the attached Appendix Tables XII and XIII.

- 1) Appendix Table XII indicates a slight improvement in compression set B with lot numbers 004 and 005. It also indicates more consistent set values with post-cured material.
- 2. Physical properties and compression set B are shown as a function of post-cure time and temperature in Appendix Table XIII. From this data, a wide range of post-cure times and temperatures are available. Physical properties and compression set B did not vary much. Tear die B showed some variation, but values were very respectable.
- 3. Extreme care must be exercised in testing and measurement of compression set B. Each ply in the plied-up samples must be individually gauged before and after testing 22 hours at 212°F. Talc must be dusted between plies to prevent blocking after compression set aging.

VI. EFFECT OF IRRADIATION

Appendix Tables XIV through XIX indicate the effects of irradiation concentration on physical properties and compression set B, for both coated and uncoated slabs. Included are tests for Shore A hardness, tensile strength, modulii, tear strength and ultimate elongation. These results are summarized below:

- A. Irradiation basically has the same effect as elevated heat aging, but of course, at much faster degradation rates.
- B. Shore A hardness, elongation and tear strength are the properties most effected.
- C. As the dosage is increased, then tensile also starts to decrease.
- D. There is essentially no difference in physical properties of uncoated vs. coated slabs.

- E. Considerable trouble was experienced in the measurement of the coated compression set B pellets due to blocking. Some problems were also experienced with the higher dosage level in the uncoated slabs. The latter could be due to surface polymer degradation.
- F. Properties of the slabs exposed in a curved position were very close whether tested in the middle or the sides. There appears to be a slightly greater effect upon the sides.
- G. No control slabs were supplied with the sets from AYO. EA slab (Appendix Table XIV) was used as a control.
- H. From this data, I would recommend exposing this product to no greater than 5 to 6 MR. Higher values will cause too much loss in elongation and tear accompanied with excessive increases in Shore A hardness.

VII. PROCESSING PARAMETERS

The following are processing parameters ultimized to produce the best optical quality Toroidal lenses at the lowest reject rate using GFM Toroidal lens mold. Modifications will have to be made depending upon size and type of mold, tooling and equipment.

A. Mixing Procedures

The following mixing procedure is recorded here as employed with our laboratory milf. This procedure differs from the Edgewood Arsenal method in that we do not use the mill guides to contain the stock. This was done to prevent stock contamination due to worn mill guides. This also may be a good method, but somewhat impractical, when larger mills are used at other facilities. In either case, each component should be milled separately before blending to eliminate a "crepe" problem associated with silicone.

- 1. Mill opening 3mm and use full cold water.
- 2. Mill 500 grams of Part A 6 times end-over-end and remove from mill in a strip.
- 3. Mill 500 grams of Part C 10 times end-over-end and remove from mill in a strip.

- 4. Open mill to 4mm.
- 5. Place Parts A and B strip together and mill end-over-end 20 times.
- 6. Remove milled stock in roll and cut preparation to weight.

Stock must not touch mill guide to prevent contamination and milling area must be thoroughly cleaned and checked using a black light as an indication.

B. Pressure (Press) Variation Trials

The press pressure was varied between 2500 and 4000 psi while keeping the transfer pot temperature at 270°F and the mold temperature at 275°F. Temperatures were measured using a surface pyrometer on the external side of the closed mold and transfer pot. Cure time, in all cases, was ten (10) minutes.

The variations in press pressure appear to effect the severity and position of flow lines in the Toroidal lens at the curing temperatures listed on the previous page. The predominate effect is due to the rate of material transfer in the mold.

Pressure, psi	Comments		
2500	Severe flow lines in the extreme sides of lens, irregular flow lines in center of lens.		
3000	Flow lines in the extreme sides and left and right bottom. Not as severe as at 2500 psi.		
3500	Slight flow lines in the extreme sides and on bottom of lens.		
4000	Slight flow lines on right and left bottom only.		

It is almost impossible to consistently eliminate the slight flow lines around the transfer gate and on the bottom side with the current design of mold. The latter are generally slight and limited to one on each side. Several solutions to this problem are listed below.

- A. Place transfer gate in top portion of lens which is not a critical optical portion of the lens.
- B. Place transfer gate outside molded lens area.
- C. Increasing the transfer gate size could possibly help, but not necessarily so.

The above recommendations should not be accomplished at this time due to Edgewood Arsenal's Toroidal lens requirements for prototype construction of complete face masks.

C. Transfer Pot Loading Variations Trials

The material charge weight in the transfer pot was varied from 65 grams to 140 grams to see the effect it had on flow lines. Normal weight used was 130 grams for silicone lot No. 005. Press pressure of 4000 psi and curing temperatures indicated in section VIII B were used.

The flow lines in the bottom (left and right sides) were evident at all transfer pot loading levels, even the partially filled lenses had them. This suggests that the positions of the transfer gate and/or type would have to be modified to eliminate these slight flow lines in the critical optical areas.

D. Transfer Pot Strata Test Trials

The ultimate effect in a molded Toroidal lens was studied when the transfer slug was composed of different colored layers of silicone. These layered slugs were loaded in the center of the pot both in a horizontal and vertical position.

When the slug was placed in the transfer pot with the layers in the vertical position, a mixing action occurred and no individual color concentration was noted.

However, when the layered slug was placed in horizontal position, definite distinct uniform layers resulted in the molded lens. Also, these layers appeared in the lens in the reverse order or position to that of the transfer slug. That is, the top color in the slug appeared on the bottom or inside of the lens while the bottom slug color appeared in the top or outside lens surface.

Additional transfer pot-loading trials were made where the charge weight was varied from 60 to 150 grams and each charge was made up of three horizontal colors with green on top, yellow in the middle and blue on the bottom.

In all cases, the colors were reversed and appeared in uniform layers in the molded lens. The reason for this appears to be that the silicone is reacting like a very viscous liquid passing through an orifice.

This phenomena should be investigated further.

E. Molding Time and Temperature Trials

1. Reduced cure times at standard molding conditions of 270°F transfer pot, 275°F mold and 4000 psi pressure for ten (10) minutes.

The cure time was decreased in increments of two (2) minutes. A two-minute cure produced a lens with insufficient cure to demold. Time of six-and four-minutes appeared to increase the number of visual optical defects in the form of flow lines.

- 2. Increased temperature trials indicated that 285°F transfer pot and 290° F mold was as high as we could go before severe molding defects, "orange peel" appeared.
- 3. The results from both 1 and 2 above were not conclusive. In order to establish optimum cure temperatures and time, it is essential that a more accurate method be employed to determine actual temperatures in the mold and transfer pot. This will be accomplished as soon as approval is granted by Edgewood Arsenal to drill holes for thermocouples and/or thermometers in the mold base.

F. Cure Reduction

Holes were drilled within 1/8" of the transfer pot and mold cavities so that we could accurately determine the actual molding temperatures via a thermocouple and/or dial thermometers. Through this we were able to determine the optimum molding conditions and minimum cycles for an acceptable Toroidal lens from this mold and press.

Conditions	Values
Pressure	3500 - 4000
Transfer Pot Temp., ^O F	290 ^O F
Mold Temp., ^o F	295 - 300
Molding Time, Min.	4 - 5
Change Time, Min.	3.5
Total Cure Cycle, Min.	7.5 - 8.5

- 1. Going to higher temperatures than indicated presents problems in precuring the elastomer and causing flow lines and "orange peel" effects.
- 2. A three minute cure could not be consistently molded over an extended period of time due to excessive mold heat loss.
- 3. When the product is undercured, the first indications are difficulty in part removal and hexagonal lines throughout the product. The latter were very hard to see with the naked eye, but were very noticeable as viewed under the shadowgraph.

Increasing the cure time helped, but an increase in mold temperature proved to be the best way cosmetically and economically.

G. Effect of Filler Levels in Silicone X4-2665

The easiest and only way available to evaluate the effect that filler concentrations have on optical, physical and processing properties would be to have Dow Corning supply us with the above polymer without any filler so that we could blend to any desired ratio.

Dow Corning confirmed our thoughts that without filler, this polymer would have very little strength and therefore, not a feasible idea. Dow further commented that they had determined the current filler level for overall optimum properties.

VIII. CONTAMINATION STUDIES

The intent of this study was to press the silicone material, after the various processes, between clear plates to determine by count the amount of contamination due to any given step. At best, this is subjective and the visual aids we tried were of little use, and in many cases, produced erroneous results and/or conclusions.

- A. The first samples were made by pressing parts A, C, and mixed A & C separately between 1 mil polyester film. This produced a hazy product.
- B. The second method was to press the same product between .100" thick clear acrylic sheets in same manner as described in "A" above. This was done both in thin sheets and .100" thick. This produced a better product, however, entrapped air proved to hinder the evaluation for contaminates.
- C. The visual aids used to determine contaminates were:
 - 1. Shadowgraph
 - 2. Projector
 - 3. Reflective Light
 - 4. Ann Arbor
 - 5. Microscope
 - 6. Naked Eye

The Shadowgraph and Projector methods could not determine the difference between air inclusions and contaminates. This was proved by viewing indicated contaminates via Microscope. These turned out to be density differences and not contaminates.

The Ann Arbor will generally indicate inclusions, but due to its smallness, it is very difficult to scan a large area.

Out of necessity, we have reviewed the plates with the naked eye and have recorded the following results. This is strictly a subjective test and it is altogether possible that some of the specks indicated only appear to be specks or that they may have been on the acrylic sheet surface. We took extreme precautions to clean the plates and keep them clean prior to pressing.

Listed in Appendix Tables XX and XXI are the results of our contaminate counts in the pressed plates and in the resulted molded Toroidal lenses.

In the March 12, 1976 run, we pressed the majority of the stock and only molded four (4) Toroidal lenses. We only had one possible contaminate and that was a wide yellowish streak which appears to be surface oriented. This could have happened in demolding.

The March 19, 1976 trials were just reversed where the majority of the stock was used to mold Toroidal lenses. From these lenses we had four (4) out of twenty-five (25) (16%) that had one very minor speck in each lens.

In both cases above, we have shown the possibility of more specks in both the virgin material and processed material than indicated in the finished product. It is our subjective judgement that the Dow Corning material is basically a very clean product. We have noticed some contaminates, but generally in isolated instances. The majority of the contaminates come from milling and in the molding cycle. We definitely know that contaminates are present in the transfer pot and are very difficult to remove due to the extremely small distance between the pot and plunger. We have also found that the more you attempt to clean the entire mold, the higher the percent of contaminates there are in the finished product.

IX. CONCLUSIONS AND RECOMMENDATIONS

A. Conclusions

1. Through this study we were able to produce acceptable X4-2665 silicone lenses that were used as prototype lenses in Edgewood Arsenal's new Protective Face Mask Program.

- 2. Although we made substantial improvements in the reject rate, it is still excessive at between 25 percent and 30 percent under optimum conditions.
- 3. Processing parameters were established which resulted in a reduction in reject rate.
- 4. High reject rate was due to contamination, flow lines and improper handling; especially of the hot molded lens.
- 5. Base silicone elastomer generally was very clean and was not a contributing factor in contamination of molded lens.
- 6. Mold and transfer pot temperatures are critical in minimizing molded flow lines.
- 7. Majority of flow lines were in the critical area and were centered around the transfer sprue.
- 8. The Toroidal lens has satisfactory optical properties when measured perpendicular to the lens surface, but has a prismatic effect in the as-worn position.
- 9. If irradiation method is used to promote coating adhesion, care must be exercised to keep the dosage low to prevent degradation of the silicone lens.
- 10. Cure times were reduced from a standard ten (10) minutes to four (4) minutes. Since the total change time was 3.5 minutes, lower cure times over an extended period were not feasible, due to excessive mold cooldown.

B. Recommendations

- 1. Further reduction in cure time can be affected by reducing the mold heat loss through heating cores in the mold.
- 2. Flow line reduction can be accomplished by proper control of molding temperatures, but consideration of placement and size of transfer sprue should be made so that if any flow lines are present, that they be in a noncritical viewing area.

- 3. The following should be accomplished to minimize rejects due to contamination.
 - a) Clean room daily, especially around mixing and molding area.
 - b) Reduce positive air flow to a minimum. Laminar air flow is recommended.
 - c) Use Clean Room type coats and caps instead of current white coats.
 - d) Use isopropyl alcohol for cleaning unvulcanized silicone processing equipment.
 - e) Use rubber gloves when mixing and handling silicone rubber.
 - f) Use lint-free cheese cloth in cleaning mold and transfer pot areas.
 - g) Use end-over-end method for mixing without touching mill guides. Thoroughly break down each component prior to blending.
 - h) Clean transfer plunger and pot thoroughly between each molding and mold, when necessary.
 - i) Eliminate unnecessary entry into room.

After thorough cleaning and prior to mixing the area should be checked with a "black light". This reveals considerable lint and contaminates throughout the processing area not seen with the naked eye. Because of this, the following procedure should be instituted:

- a) All areas should be cleaned with a damp cheese cloth using the "black light" to insure thorough cleaning.
- b) Floor should be damp-mopped.

- 4. Since the silicone attracts foreign material statically, the area should be kept moist to keep this problem to a minimum.
- 5. Viewing portion of lens should be redesigned to eliminate the as-worn optical problems.
- 6. Strata programs should be established to determine the feasibility of transferring a uniform layer of a dissimilar material for better barrier properties.

Table A-1

-XPOSURE

(2)	(3)	\$ =	3(1)	2F 2R	1F (2)	Exposure Time, Hrs. Sample No.
Coating started to craze on exposed surface only at 100 hours Increase in haze after washing (220 hours) due probably to more light being deflected	Turned yellow after 30 hours.	91.2 91.1	92.0	88.2 88.3	87.3 87.3	1LT.8
raze on exp er washing	30 hours.	3.50 3.65	4.52	5.81 6.09	5.21 5.26	Haze
osed surface (220 hours		91.0 90.9	86.8	88.8 88.7	84.6 87.4	100 TLT, 8
ce only at 100) due probab		5.86 6.97	17.87	5.79 6.81	11. 48 5.73	Haze, 8
hours ly to more li		90.3	84.1	88.9 8.6	84.3 86.3	TLT, %
ght being de		7.22 7.14	25.8	6.00 7.00	12.45 7.58	220 Haze, &
flected.		89.2 91.6	84.7	91.5 89.2	82.0 83.4	
		4. 98 3. 90	21.6	3. 82 5.40	28.4 24.3	(Lightly Washed)

Appendix A

Sample Number

Sample Identification

Upjohn coated Toroidal Lens Wilmington Chemical coated Toroidal Lens Upjohn 2363-80A film Control Toroidal Lens - uncoated

Table A-2

TOROIDAL LENS - OPTICAL TESTS

REF. FIG. 1 MIL-L-38169

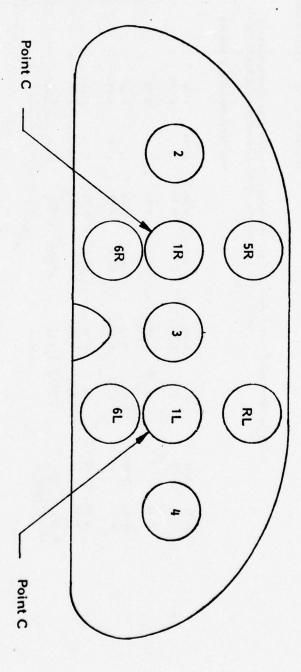


Table A-3
ENS - OPTICA

	MIL-L-38169 Specification
	Material:
Lot 32059	Material: Dow Corning Silicone

Haze, §	Total Light Trans, &	Ann Arbor	Defination (Lines Resolved)	Spherical & CLN Power	Refractive Power	Prismatic Deviation	Test Position Test
4.54	91.8	22	* *	Not Re	065	. 02	155
		22	80 28	quired in	+.06 055	. 07	2R Right
		22	34	Above	+.03	. 10	1%
		Rej.	56	Spec., Bu	01		199
		OK Rej.	56	Not Required in Above Spec., But Will Be Tested In Future.	÷.04	. 08	Center 3
4.80	91.6	OK Rej.	28	d in Futur	+.03 +.065	.08	l#
		Rej.	880	Ģ	005 +. 05	.05	#
		Rej.	56 56		02 03	.07	SL SL SL
		Rej.	8 6		06	.07	<u> </u> 6
Relaxed	Relaxed	Relaxed Fixture	Relaxed Fixture		Relaxed Fixture	Relaxed Fixture	Tested

Table A-4

TOROIDAL LENS - OPTICAL EVALUATION

MIL-L-38169 Specification

-38169 Specification Material: Dow Corning Silicone
Lot 001

Haze, %	Total Light Trans, &	Ann Arbor	Defination (Lines Resolved)	Spherical & CLN. Power	Refractive Power	Prismatic Deviation	Test Position
3.65	91.2	읒읒	56 80	Not Re	01 +.065		175
		읒읒	80	quired Ir	+.03	2 2	Right 5
		읒읒	56 80	Above :	+.02 01	 8 9	SR SR
		읒읒	8 8	Spec., Bu	+.015	. 95	15
		R OK	56 80	Not Required In Above Spec., But Will Be Tested In Future	01 +.04	. 02	Center 3
3.59	91.1	22	8 8	ed in Futur	+. 045 045	.07	I=
		Rej.	8 8		+.06		₽
		읒읒	8 8		+.06	. 07	15. ₹
		OK Rej.	88		+ + 06 + 05	. 02	 €
Relaxed	Relaxed	Relaxed Fixture	Relaxed Fixture		Relaxed Fixture	Relaxed Fixture	Tested

Table A-5

!	
	MIL-L-38169 Specification
	Material:
Lot 002	Material: Dow Corning Silicone
	ro .

Haze, % 3.94	Total Light Trans, \$ 91.0	Ann Arbor OK Rej.	Defination 80 (Lines Resolved) 80	Spherical & CLN. Not 9 Power	Refractive +.04 Power +.06	Prismatic .07 Deviation .05	Test	Test Position 1R
		♀♀	8 8	Required In	+.03	. 06		Right 5
		22	56	Above S	.05			5R
		22	68 80	pec., But	+.015 +.05	. 03		 \$
		22	8 8 8	Not Required in Above Spec., But Will Be Tested In Future.	035 +.05	. 04		Center 3
3.99	90.9	22	80	In Future	+. 005 055			
		Rej.	80 80		+.015	. 06		# [
		Rej.	80 80		·.01	.07		5 ₹
		OK Rej.	S 80		+. 055	.07		ę.
Relaxed	Relaxed	Relaxed Fixture	Relaxed		Relaxed Fixture	Relaxed Fixture		Tested

Table A-6

TOROIDAL LENS - OPTICAL EVALUATION

MIL-L-38169 Specification

Material: Dow Corning Silicone

Lot 003

Haze, %	Total Light Trans, &	Ann Arbor	Defination (Lines Resolved)	Spherical & CLN. Power	Refractive Power	Prismatic Deviation	Test Position
4.28	90.7	읒읒	56 80	Not Re	+.01	. 04	۱ ۶
		22	8 8	quired I	+.005	. 06	2R Right
		R OK	8 8	n Above	÷.01	. 04	X SR
		<u> </u>	8 8	Spec., E	00		18
		22	8 8 8	Not Required In Above Spec., But Will Be Tested In Future	+. 01 +. 045	. 0.04	Center 3
4.32	90.6	99	80	ed In Futu	00 +. 06	. 06	۱≓
		Rej.	8 8	7	+. 005	. 05	#
		Rej.	8 8		+.015	. 04	Left SL
		Rej.	8 8		+.01		lę.
Relaxed	Relaxed	Relaxed Fixture	Relaxed Fixture		Relaxed Fixture	Relaxed	Tested

Table A-7

TOROIDAL LENS - OPTICAL EVALUATION

MIL-L-38169 Specification

Material: Dow Corning Silicone Lot 004

Horizontal Vertical	Horizontal Vertical	Prism (Expressed in vertical and horizontal deviation)	Haze, &	Total Light Trans, &	Ann Arbor	Defination (Lines Resolved)	Refractive or Spherical Power	Prism	Test	Test Position
+.03	+.04		3.27	91.0	읒읒	80	+.04 035	09		ΙĦ
+. 02 10	+ . 04				읒읒	8 8	+.01			Right 2R
-,02 -,12	06 12				Rej.	8 8	+.02	. 12		
+ .04 02	+. 02				Rej.	800	+.015 025	. 09		18
÷.02	- + . 03 10				Rej.	8 8	+. 01 065			Center 3
· . 08	+.05		3.17	91.0	읒읒	80	+.035	. 12		F
+ . 09	+ + 06				Rej.	8 8	025 +. 045	= :		ا د ا2
+.05 11	- + . 06 - 08				웃옷	8 8	005	. 12		ST ST
	06 +. 12				OK Rej.	80 80	+.02 06	. 08		 \$
Fixture	Relaxed		Relaxed	Relaxed	Relaxed Fixture	Relaxed Fixture	Relaxed Fixture	Relaxed Fixture		Tested

Table A-8

Toroidal Lens Comparison

Total Light Trans. & Haze - Before & After Fade-o-meter Aging

Lightly Washed TLT, 8 Haze, 8	As Is TLT, % Haze, %	After 600 hrs. in Fade-o-meter	Original TLT, %	Lot No.
92.4 4.69	89.6 13.58	ade-o-meter	91.7 4.67	X32059
91.2 4.79	87.5 20.95		91.1 3.62	00
91.1 4.59	89.0 13.85		91.0 3.96	002
91.1 4.61	88.9 15.08		90.6 4.30	003
91.6 3.04	89.7 11.97		91.0 3.22	004
11	11		90. <i>7</i> 3.53	005

Table A-9

ASTM Slabs - Physical Property Comparison
No Post Cure vs Post Cure

Lot No.	X32059	001	002	003	004	005
No Post Cure			1	1		1
Shore A Hardness	50	53	53	58	58	!
100% Mod., psi	195	240	200	265	250	-
2008 Mod., psi	395	490	405	530	530	-
300% Mod., psi	630	785	620	785	805	-
Tensile, psi	1045	1135	1060	1170	1180	1
Elongation, %	470NB	044	485NB	480NB	450NB	1
Tear, Die "C"	182	124	252	243	140	-
Post Cure						
Shore A Hardness	61	56	61	62	49	-
100% Mod., psi	305	275	250	360	385	-
200% Mod., psi	645	620	555	745	740	i
300% Mod., psi	1034	970	870	1065	1045	!
Tensile, psi	1315	1185	1305	1280	1210	i
Elongation, %	395	370	470	380	360	-
Tear Die "C"	126	175	158 8	2	116	
				•	;	

Press Cure : 101/2700F Oven Post Cure: 4 hrs./3500F

Table A-10

Toroidal Lens - Physical Property Comparison
No Post Cure vs Post Cure

Press Cure : 101/270°F Oven Post Cure: 4 hrs./350°F	Tear Die "C"	Elongation, %	Tensile, psi	300% Mod., psi	200% Mod., psi	100% Mod., psi	Shore A Hardness	Post Cure	Tear Die "C"	Elongation, %	Tensile, psi	300% Mod., psi	200% Mod., psi	100% Mod., psi	Shore A Hardness	Lot No. No Post Cure
10'/270°F 4 hrs./350°F	124	370	1085	1085	725	275	62		153	500NB	1315	705	420	160	53	X32059
	117	410	1200	885	540	280	57		240	490NB	1050	540	330	145	47	001
	123	395	1265	955	645	290	60		245	480NB	1200	690	425	200	56	002
	132	365	1315	1125	730	375	63		224	480NB	1225	765	515	250	55	003
	Ξ	345	1240	1095	740	325	62		156	450NB	1200	825	530	240	55	004
	118	385	1200	945	665	305	62		174	490NB	1155	655	415	200	52	005

Appendix A

Table A-11

TOROIDAL LENS COMPRESSION SET EVALUATION

(No Post Cure vs. Post Cure - 22 Hrs. @ 212°F)

005	004	003	002	001	X32059	Lot Number
31.13	18.1	35.7	34.7	36.25	21.33	No Post Cure*
13.49	11.36	17.15	17.28	15.19	12.35	Post Cured**

^{*}Lens Cured 10 minutes @ 270°F **Post Cure 5 hours @ 350°F

Table A-12

Post Cured Silicone Toroidal Lenses

Effect on Physical Properties

7. 7.	9.	8 7 6 5	+ 3	2 .	Set No.
1/4000	1/3750 2.5/3750	1/3500 2.5/3500 3.5/3500 5/3500	2.5/325 ⁰ 5/325 ⁰	N.P.C.	Post Cured Hrs/OF
60	59	60	60	60	Shore A Hardness
390	385 575	430 410 405	410 425	405	100%
790 830	765 790	800 785 860 770	780 805	760	2008
1075	1110	1080 1100	1130 1135	1040	300%
No	Break 320	On All S	Samples		Tensile & Elongation
170.3 213.9	172.2 170.5	161.6 206.4 187.0 145.3	211.9 155.6	182.3	Tear Die B
12.0 14.4	10.4 12.9	16.5 11.9 12.4 11.2	16.8 13.5	25. 8 14. 0	Compression Set B 22 Hrs/2120F.

Gentex Dies
Lot No. 005
Press Cured - 51/2950F

Table A-13

EFFECTS OF IRRADIATION ON SILICONE SLABS

INITIAL SLABS FROM EA

Physical Properties				
Identification	EA-Control	(17-20 Mega-Rads EA-Irrad	AYO Irrad	- 3/26/76 4
Shore "A" Hardness	62	78	78	77
100% Modulus, psi	325	_	_	
200% Modulus, psi	710	_	_	
300% Modulus, psi	1075	7	_	
Tensile Strength, psi	1200	1165	1080	980
Elongation, %	330	95	85	90
Compression Set B				
22 hours @ 212°F, %	20.3	13.9	8.7	10.7
Blocking				,
After Compression Set 22 hours @ 212°F	Slight	Moderate	Severe	Severe
Blocking, ASTM				
D854-48 After 24 hours @ 1600F 2 kg. wt. on 4"x1" Samples	None	None		

Uncoated Silicone Slabs Table A-14

Irradiation Effect On Physical Properties

				=												
	30.8	28.6	26.4	24.2	22.0	19.8	17.6	15.4	13.2	11.0	8.8	6.6	4.4	2.2	0	MR
	78	78	75	75	75											Shore A Hardness
							885	915	865	675	645	465	465	375	325	Modulii, Psi 100% 200%
												1140	920	815	710	2008
	485	505	625	, 665	750	880	915	940	1170	1260	1230	1190	1250	1295	1200	Tensile St.
	30	30	50	60	60	80	100	110	140	170	190	210	265	320	300	Elong., 8
	17	33	29	30	40	35	56	49	52	58	62	81	125	140	1	Tear Die B
	30	15	#2	46	47	45	#	56	63	77	77	90	102	107	1	Psi Die C
	14.0	12.3	11.5	12.7	17.0	12.0	13.9	13.7	13.5	14.7	11.6	11.6	10.7	11.5	20.3	Compression Set B 22 Hrs/2120F.
)(endi	x A							42							

App

*No Control For This Group - These From Original Slabs Sent by EA For First Irradiation Tests. (See Table A-13)

Table A-15

Coated Silicone Slabs

14.	13.	12.	=.	10.	9.		7.	6.	5.		ω	2.	-	Set No.	
30.8	28.6	26.4	24.2	22.0	19.8	17.6	15.4	13.2	11.0	8.8	6.6	4.4	2.2	I _R	
80	80	89	78	75	77	75	73	73	72	70	68	67	65	Shore A Hardness	
							900	825	775	695	550	475	395	Modulii, Psi 100% 200%	Irradia
											1225	1030	835	11, Ps1 2008	ation Effec
405	350	450	465	645	685	785	965	1080	1250	1285	1235	1235	1265	Tensile St.	Irradiation Effect On Physical Properties
30	30	30	50	60	60	85	100	125	165	200	215	245	300	Elong., %	operties
24	27	42	34	34	ŧ	40	47	62	51	72	90	103	128	Tear, Die B, %	
11.5		•	•	4.1	•	5.2	•	•	,	9.4		7.2	•	Compression Set B 22 Hrs/212°F.	

Table A-16

Uncoated Silicone Slabs Curved

(Side)

14.	13.	12.		10.	9.	.00	7.	6.	5		3.	2.		Set No.	
30.8	28.6	26.4	24.2	22.0	19.8	17.6	15.4	13.2	11.0	&	6.6	4.4	2.2	NR NR	
82	81	81	81	80	79	78	77	76	73	72	69	68	65	Shore A Hardness	1=
					1300	1230	1100	920	1050	780	670	670	455	1008	rradiation
										1470	1300	1235	955	2008	Effect On P
670	930	755	785	970	1230	1230	1270	1335	1505	1450	1505	1460	1520	Tensile St., Psi	Irradiation Effect On Physical Properties
65	80	65	70	75	100	100	115	150	150	190	230	255	330	Elong., &	88
33.8	42.0	47.2	49.4	59.8	88.1	55.7	76.2	65.7	87.7	72.1	140.9	172.0	150.2	Tear Die B	

Table A-17

			Uncoate	Uncoated Silicone Slabs Curved	abs Curved		(Middle)
			rradiation	Effect On Phy	Irradiation Effect On Physical Properties	les .	
Set No.	N.	Shore A Hardness	1008	200%	Tensile St., Psi	Elong., %	Tear Die B
	2.2	65	460	875	1355	320	175.3
2.	4.4	67	585	1130	1380	245	145.1
'n	6.6	69	580	1200	1405	235	138.8
•		72	680		1440	185	104.2
.5	11.0	73	825		1435	175	82.8
6.	13.2	75	935		1320	140	65.7
7.	15.4	77	1020		1180	110	76.1
	17.6	78	1130		1130	100	47.3
9	19.8	78			1085	90	64.6
10.	22.0	79	1170		1170	100	54.4
1.	24.2	81			835	77.5	46.8
12.	26.4	81			710	70	47.0
13.	28.6	82			550	65	31.2
7.	30.8	82			640	70	52.6

Table A-18

Viton A Additive Silicone Slabs

21.	20.	19.	18.	17.	16.	15.			21.	20.	19.	.	17.	16.	15.	Set No.	
28.6	(No B)	19.8	15.4	11.0	6.6	2.2			28.6	24.2	19.8	15.4	11.0	6.6	2.2	MR	
81	•	78	75	73	70	64			82	79	78	76	72	70	64	Shore A Hardness	_
		1310	960	940	720	555	rradiation	Viton .	ı	ı	1150	1030	ı	710	465	100%	rradiation
					148	1020	Effect (A Additi						1125	940	2008	Effect (
						1415	Irradiation Effect On PhysicI Properties	Viton A Additive Silicone Slabs								3008	On Physical
1070		1365	1415	1390	1438	1400	Properties	Slabs	975	1150	1235	1185	450	1125	1330	Tensile St.,	Irradiation Effect On Physical Properties
75		105	145	155	200	285			85	90	105	110	75	200	275	Elong., %	
31.9		58.5	63.1	64.5	102.1	130.4			52.6	31.9	53.1	47.3	43.0	134.4	184.7	Tear Die B	

Table A-19

MATERIAL CONTAMINATION STUDY

	Identification	Number of Specks
A.	Pressed Between 1 Mil Polyester Film	
	1. 10-11-004 A	0
	2. 10-11-004 C	0
	3. 10-11-004 A & C (1st. Batch)	1
	4. 10-11-004 A & C (2nd. Batch)	. 1
В.	Pressed Between .100" Acrylic Clear	
	Sheets - 3/12/76	
	5. 005 A	1
	6. 005 A	1
	7. 005 C	. 0
	8. 005 C	4
	9. 005 C (No Wash)	1
	10. 005 A & C	0
	11. 005 A & C (170 Grams)	0
	12. 005 A & C	0
	13. 005 A & C	0
c.	Pressed Between .100" Acrylic Clear	
	Sheets - 3/19/76	
	14. 005 A	5
	15. 005 C	1
	16. 005 A & C	1
	17. 005 A & C	3

NOTES: Thin Sheets - 60 grams stock used .100" Thick Samples - 100 grams stock used

Table A-20

Toroidal Lens Contamination Study

Identification	Remarks
3-12-005-1	Good
3-12-005-2	Scuff Marks & Flow
3-12-005-3	Good
3-12-004-4	Yellow Line
3-19-002-1	Good
3-19-002-2	Flow Line
3-19-002-3	Good
3-19-002-4	Brown Speck
3-19-005A-5	Good
3-19-005A-6	Flow Lines
3-19-005A-7	Good
3-19-005A-8	Scuff Mark
3-19-005A-9	Good
3-19-005A-10	Scuff Mark, Flow Line
3-19-005B-11	Black Speck
3-19-005B-12	Scuff Mark, Flow Lines
3-19-005B-13	Good
3-19-005B-14	Good
3-19-005B-15	Scuff Mark, Flow Lines
3-19-005B-16	Scuff Mark, Brown Speck
3-19-002-17	Flow Line
3-19-002-18	Good
3-19-005C-19	Short Flow Line
3-19-005C-20	Good
3-19-005C-21	Black Speck
3-19-005C-22	Scratch & Scuff Marks
3-19-005C-23	Good
3-19-005C-24	Good
3-19-005C-25	Good



Memorandum

Omnitech Division

to:

John Daugherty

from

Albert J. Laliberte

refer to

date: March 8, 1976

subject :

"Edgewood Arsenal Lenses"

In your memo of Jan. 7th, you asked for "Cylinder Power" readouts on 3 toroidal Edgewood Arsenal Lenses. These were one control lens from Edgewood (EA-2), and two lenses molded by you identified as 12-17-003A-3 and 12-19-002B-13 (reject).

First of all, I measured these in a Lensometer. This instrument shows negative
power at a magnitude of about - .12
diopter, with an image fuzziness indicating
a degree of "Cylinder", which, however, could
not be resolved accurately; or significantly
measured on the Lensometer.

Since the lens is essentially plano, I measured the power on our telescope. All lenses measured the same.

May I point out, John, that the minus readings observed are inherent in the design of this lens and are to be expected.

The .06 "Cylindrical Power" encountered, in my opinion, is at the acceptable limit for such products as safety lenses and shields; in fact, I consider this very good for this product.

None of the above relates to the most bothersome aspect which will be encountered wearing this lens, which is prismatic image displacement due to positioning of the lens as worn. This is a completely different effect than the measurements described above.

We are returning your lenses.

Sincerely,

OMNITECH, INC. (Subsidiary of GenTex Corp.)

A.J. Laliberte

AJL:ca

Enclosures: 1 Copy your memo Jan. 7th

Lenses

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